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**FEMTOSECOND PHOTOACOUSTIC EQUIPMENT FOR
MATERIALS CHARACTERIZATION**

AFOSR Award No.F49620-02-1-0276

Final Report

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EXECUTIVE SUMMARY

The objective of this project was to configure non-contact nondestructive photo-acoustic measurement systems for characterization of micro- and nano-structured material systems including thin films and coatings. Specifically, a femtosecond photo-acoustic materials characterization facility has been established. At Northwestern University, we are actively pursuing research efforts to increase the sensitivity of photo-acoustic metrology. These efforts have led to advances in laser-based ultrasonics, fiber-optic ultrasonic sensors, and photo-acoustic characterization of thin films. The femtosecond photo-acoustic system has now extended our acoustic frequency reach to the Gigahertz range. Specific research projects that we are continuing to pursue include: transient grating and pump-probe photo-acoustic experiments on very thin films, MEMS devices, and nano-structured composites. Specific material and geometric properties that can be characterized using the photo-acoustics facility include: elastic moduli, thickness, and residual stress.

NU has one of the leading photo-acoustic and ultrasonics materials characterization research programs in the nation. The femtosecond laser equipment will provide our graduate students and faculty the opportunity to extend this research program to the area of very high-frequency photo-acoustic materials characterization of micro- and nano-dimensioned structures.

Some of the modes of operation of the femtosecond photo-acoustic system have already been demonstrated. The applications phase of this project will continue to be conducted in parallel with other ongoing projects at the Center for Quality Engineering at Northwestern University. Specific applications of the proposed system include characterization of diamond-like carbon coatings on substrates (ongoing NIST supported project on "Photoacoustic Characterization of DLC Coatings") as well as characterization of free-standing thin films (ongoing NSF supported project on "Laser Ultrasonic Characterization of Coatings").

I INTRODUCTION

Micro- and nano-structured materials are rapidly transitioning from the laboratory to real world systems. Thin film technology plays a major role in microelectronic, photonic, and micro-electromechanical systems (MEMS), as well as in wear protection and thermal barrier coatings (TBCs). Novel sensors are now fabricated using MEMS technology. Advanced lightweight composite materials can now be built from the bottom up using nanotubes and nanowhiskers to obtain high modulus and strength. Many of these technologies are of direct relevance to the Department of Defense: avionics (MEMS, microelectronics); engines (TBCs, wear coatings); structures (high performance nano-composites); and nondestructive health monitoring (MEMS sensors). These technologies are not only intended for newer generation aircraft and ship structures, but many of these can also be retro-inserted into the existing fleet to increase performance or to extend lifetime.

The development of micro- and nano-structured materials has necessitated the development of advanced measurement techniques to characterize their properties. It is well known that the properties of bulk material are not necessarily indicative of the properties of the corresponding micro- and nano-structured materials. At these scales, the mechanical properties are typically investigated using scratch and nano-indentation tests or through Brillouin scattering and atomic force microscopy. Such techniques are either destructive or are not suited for in situ testing. *The primary objective of this proposed project was to configure non-contact nondestructive **photo-acoustic** measurement systems for materials characterization of micro- and nano-structured material systems.* Photo-acoustic metrology uses pulsed laser irradiation to nondestructively induce very high frequency ultrasound in these structures via rapid thermal expansion. The resulting ultrasonic wavepackets are also measured in a non-contact nondestructive manner using interferometric or diffractive optical probes.

High-frequency photo-acoustic interrogation techniques can provide information about the mechanical, thermal and electronic properties of materials, and our primary interest at NU is in their mechanical properties. Optical measurement systems enjoy several advantages:

- they are non-contact and nondestructive (unlike nano-indentation techniques for instance), leading to increased speed of inspection;
- they can be used for *in situ* measurements, unlike typical atomic force microscopy, Brillouin scattering, or SEM techniques;
- they are couplant independent (unlike acoustic microscopy techniques), providing absolute measurements of ultrasonic displacements;
- they have a very small footprint and so can be operated on curved complex surfaces;
- they are broadband systems providing information from the kHz to the GHz range, enabling the probing of macro-structures to very thin films.

In order to probe the properties of micro- and nano-structured material systems, we have configured a femtosecond photo-acoustic system as described below.

II Instrumentation Acquired

A. Femtosecond laser source:

- (a) a refurbished Argon-ion 5W CW pump source laser at 514nm;
- (b) a refurbished mode-locked Ti:Sapphire laser that is pumped by (a) to produce ~100 fs pulses at 750-900nm with a rep rate of 80MHz;
- (c) a refurbished 5kHz Nd:YLF intra-cavity frequency doubled laser to pump a regenerative amplifier;
- (d) a new regenerative amplifier which uses the output of (b) to produce ~1mJ, ~130fs pulses at a rep rate of 1-5kHz;

These items were purchased from Spectra-Physics as a system for \$195,000. Some of the components purchased were refurbished systems (with full “new purchase” warranty of one year) in view of the reduced level of funding allocated to this project. For the same reason, second and third harmonic generation crystals, originally included in the proposal, were not purchased.

B. Characterization and detection components

- (e) an automated optical delay line for pump-probe experiments;
- (f) a lock-in amplifier for phase-sensitive detection;
- (g) a USB2000 spectrum analyzer

An autocorrelator and power meter included in the original proposal to characterize the femtosecond laser pulses, were not purchased. The cost of the above components as nearly \$5000.

The total cost of the system was about \$200,000 including shipping charges. The system that has been configured is slightly different from the one in the proposal in order to fit the available funds. This was accomplished by eliminating some components, and using some refurbished components. However, the system as configured has most of the functionality of the originally proposed system.

A schematic of the femtosecond photo-acoustic system is shown in Fig. 1. The laser source consists of an argon-ion laser whose output pumps a mode-locked Ti:Sapphire system to produce femtosecond laser pulses. These are passed through a regenerative amplifier stage to produce $\sim 1\text{mJ}$, $\sim 130\text{fs}$ pulses at 1 kHz rep rate.

III. RESEARCH ACCOMPLISHMENTS

For photo-acoustic metrology, the femtosecond laser output is used in an optical setup that allows both transient induced grating measurements as well as pump-probe measurements. The bulk of the laser energy is used for acoustic generation in the test sample. The laser beam is split in two and recombined at the test sample to provide an array ('grating') thermoelastic source which generates high frequency ultrasound by rapid heating and expansion. This kind of a source launches bulk acoustic waves at several tens of GHz that propagate through the thickness of the structure, and on a longer time scale lead to guided surface acoustic waves on the order of 1-5GHz. The bulk acoustic waves can be used to interrogate the through-thickness properties of very thin films and coatings using a standard pump-probe approach. This is done by using a (very weak) part of the femtosecond laser beam itself as a probe beam to interrogate the acoustically-induced deformation and change in optical properties of the test sample. The femtosecond probe pulse provides a snapshot of the time evolution of the acoustically-induced signal.

By obtaining a sequence of these snapshots (by adjusting the optical delay between the arrival of the pump and the probe pulse using the optical delay line in Fig 1), a time history of the acoustically-induced deformation and change in optical properties can be reconstructed over several picoseconds. On a somewhat longer time scale, surface or guided acoustic waves develop, and these can be interrogated by a diffractive or interferometric optical technique. Here, in lieu of the femtosecond probe pulse, a separate laser beam is guided to the sample for detection.

Specific applications of the proposed system include characterization of diamond-like carbon coatings on substrates (ongoing NIST supported project on "Photoacoustic Characterization of DLC Coatings") as well as characterization of free-standing thin films (ongoing NSF supported project on "Laser Ultrasonic Characterization of Coatings"). These efforts are ongoing.

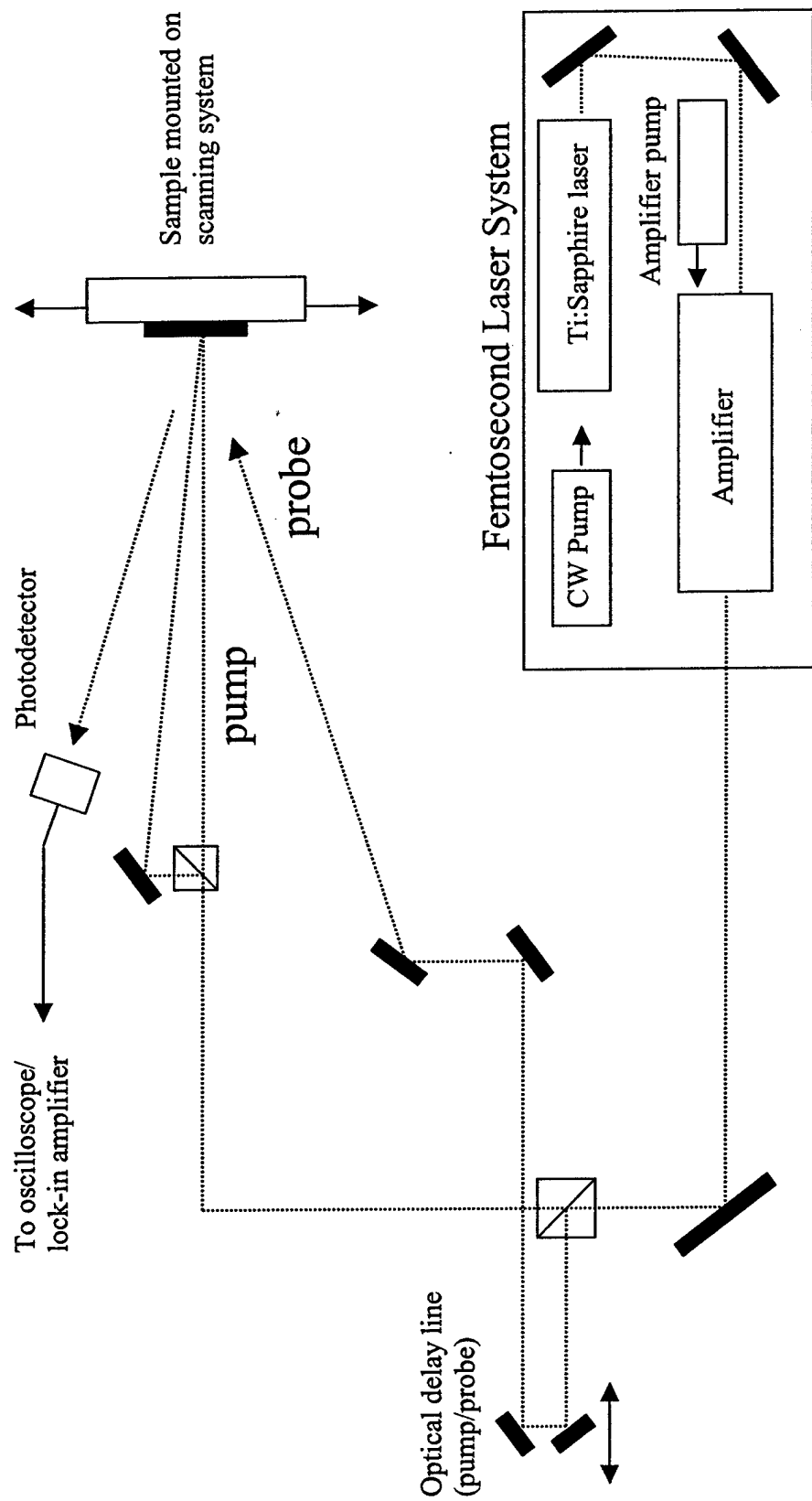


Figure 1: Femtosecond Photo-acoustic equipment

IV. PERSONNEL SUPPORTED

None. This is an equipment proposal. However, graduate students Feifei Zhang and Zhou Yi are involved in ongoing experiments using this setup.

V. PUBLICATIONS

These are some of the publications of the PI that are closely related to this proposal.

[1] C.M. Hernandez, T.W. Murray, and Sridhar Krishnaswamy, "Photoacoustic Characterization of the Mechanical Properties of Thin Films," Applied Physics Letters , vol. 80, No. 4, 2002.

[2] Zhou Yi, Todd W. Murray and Sridhar Krishnaswamy, "Photoacoustic imaging of surface wave slowness using multiplexed two-wave mixing interferometry," in IEEE UFFC, vol. 49, No. 8, p1118-1123, (August 2002)

VI. HONORS/AWARDS:

The PI was invited to make presentations on laser ultrasonics and applications to thin film characterization at the joint KSME-KSNT conference held in Busan, S. Korea, Aug 22, 2002, and at the Gordon Research Conference on Photothermal and Photoacoustic Phenomena held in New London, CT, June 2003.

VII. TRANSITIONS:

Ongoing collaboration with Caterpillar on laser ultrasonics applications to nano-structured DLC coatings.

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